

Training **Identification of Heroin**

I. Introduction:

Heroin samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS.

II. Reagents:

- A.) 9:1 Methylene Chloride/Isopropanol or Ethanol.
- B.) Methanol (solvent rinse for GC)
- C.) 0.1 N HCL: add 8.3 mL concentrated HCL to sufficient water to make 1 L. (quantification).
- D.) Benzopinacolone and 1.0 mg/mL heroin (quantitation standard).
- E.) 10% K₂HPO₄: dissolve 10g K₂HPO₄ in sufficient water to make 100 mL. (quantification).
- F.) Benzopinacolone in Methylene chloride (quantification internal standard).
- G.) Anhydrous Sodium Sulfate (Na₂SO₄).

III. Equipment:

- A.) Analytical balance
- B.) Weigh paper
- C.) Pipettes
- D.) 25 mL volumetric flask
- E.) Stoppered test tube
- F.) 2 mL autosampler vials with Teflon caps
- G.) GC/FID: HP 6890 or HP 5890 series
- H.) GC/MS: HP 5890/5972 or HP 6890/5973 series.

IV. Procedure:

A.) Chromatography by GC/FID and GC/MS.

1. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
2. Add 1-2 mL of Ethanol or 9:1 Methylene Chloride/Isopropynol to the vial and cap.
3. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples, Standard.
4. GC/FID conditions are as follows:
Method: EXP.M

Oven:

Initial Temp: 245°C
Initial Time: 0.00 min.
Rate: 10°/min.
Final Temp: 290°C
Run Time: 10 min.
Max. Temp: 325°C
Equilibration Time: 0.5 min.

Inlet:

Mode: split (35:1)
Initial Temp: 250°C
Pressure: 24.99 psi
Gas Type: Helium

Column:

Capillary: HP-1 30m x 320um
Initial Flow: 3.3 mL/min.

Detector:

Temp: 300°C
Hydrogen Flow: 30.0 mL/min.
Air Flow: 400 mL/min.
Makeup Gas: Helium

5. Obtain chromatographs. If heroin is present, the instrument will detect a peak with a retention time around 4.5 minutes and will generate a report with accompanying chromatograph.
6. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.
7. Place same sequence on the GC/MS autosampler and run.
8. GC/MS conditions are as follows:
Method: EXP.M

Oven:

Initial Temp: 230°C
Initial Time: 0.00 min.
Max. Temp: 325°C
Equilibration Time: 0.50 min.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 10 min.

Inlet:

Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi

Gas Type: Helium

Column:

Capillary: HP-1MS 25m x 200um x 0.33um

Max. Temp: 300°C

Initial Flow: 1.0 mL/min.

9. If heroin is present in sample, the instrument will detect a total ion peak at approximate retention time of 7.2 minutes and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance (see graph, last page).

B.) Quantitative Procedure:

1. Extract sample by weighing out 100 mg of sample in a 25 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.
3. The heroin standard is prepared and should contain about 1.0 mg/mL of Benzopinacolone and 1.0 mg/mL of heroin.
4. The internal standard is Benzopinacolone in Methylene Chloride.
5. In a stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K₂HPO₄. Two layers will form in tube.
6. In a labeled autosampler vial, add enough sodium sulfate (Na₂SO₄) to cover the bottom of the vial.
7. From the sample test tube, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Heroin Standard (inj.#1), Heroin Standard (inj.#2, Calibration), Heroin Standard (inj.#3), Blank, Sample(s), Heroin Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

V. Results:

- A.) Heroin is reported as positive when GC and GC/MS retention times and spectra match standard Heroin.
- B.) Record results of the GC and the GC/MS in the logbook. Then transfer the results to appropriate sample cards that came with

the actual samples. Include date of analysis, results and Chemist initials.

- C.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.